United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program

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Title: Reporting Results		
Revision: 8	Replaces: 06/01/02	Effective: 07/01/03

1. Purpose:

To provide standard procedures for reporting results to the USDA/AMS Pesticide Data Program (PDP).

2. Scope:

This standard operating procedure (SOP) shall be followed by all laboratories conducting residue studies for PDP, including support laboratories conducting stability or other types of studies that may impact the program.

3. Outline of Procedures:

5.1 Determination of Residue Concentrations for PDP Reporting Purposes
5.2 Reporting Non-violative Results
5.3 Reporting a Presumptive Tolerance Violation (PTV)

4. References:

- PDP QA/Technical Meeting, May 7-9, 2003
- USDA/AMS PDP QA/Technical Meeting, April 9-11, 2002
- USDA/AMS PDP QA/Technical Meeting, February 21-22, 2001
- USDA/AMS PDP QA, April 4-5, 2000
- SOP PDP-DATA-02, Attachment 01, PDP Laboratory Information Form (LIF) Codes, March 2003
- USDA/AMS PDP QA Meeting, May 18-20, 1999
- U.S. EPA, Reporting of Study Results, 40 CFR part 160.185
- U.S. EPA, Tolerances and Exemptions from Tolerances for Pesticide Chemicals in or on Raw Agricultural Commodities, 40 CFR part 180

5. Specific Procedures to be Followed:

This SOP represents minimum PDP requirements and is presented as a general guideline. Each laboratory shall have written procedures that provide specific details concerning how the procedure has been implemented in that laboratory.

5.1 Determination of Residue Concentrations for PDP Reporting Purposes

a. Validated Pesticide/Commodity Pairs

A pesticide/commodity pair is considered validated when all applicable modules in SOP PDP-QC-07 have been met.

1. Compounds appearing on the analytical results list for which results are not/cannot be reported shall be coded as "M" [not analyzed (e.g., compound not screened in applicable run)] or "U" [unable to analyze (e.g., associated matrix spike failed)] in the mean result field of the LIF.

2. Do not report residue concentrations less than the verified LOD. These results shall be coded as "ND" (non-detect); "NA" (non-detect – averaged analysis); or "NR" (non-detect – rerun analysis) in the mean result field of the LIF.

3. Numeric concentrations below the LOQ are considered low confidence values associated with a qualitative finding. A concentration value is not required when a pesticide is detected at or above the determined LOD and below the determined LOQ. The laboratory must code the finding as "Q" [residue at below quantifiable level (BQL)] in the "Annotated Info." section of the LIF. The concentration will be converted to ½ LOQ in the PDP database for reporting purposes. Detections shall be coded as: "O" (detect – original analysis value); "A" (detect – average of original and re-extract); or "R" (detect – re-extraction analysis value) in the mean result field of the LIF. The laboratory shall enter "H" (standard not in matrix);

"M" (standard in matrix); "PH" (standards prepared using analyte protectants - not in matrix); "PM" (standards prepared using analyte protectants - in matrix); "SH" (internal standards - not in matrix); or "SM" (internal standards - in matrix) in the quantitation field of the LIF.

4. Residue concentrations greater than or equal to the LOQ shall be reported on the LIF. Detections shall be coded as: "O" (detect – original analysis value); "A" (detect – average of original and re-extract); or "R" (detect – re-extraction analysis value) in the mean result field of the LIF. The laboratory shall enter "H" (standard not in matrix); "M" (standard in matrix"PH" (standards prepared using analyte protectants - not in matrix); "PM" (standards prepared using analyte protectants - in matrix); "SH" (internal standards - not in matrix); or "SM" (internal standards - in matrix) in the quantitation field of the LIF.

b. Unvalidated Pesticide/Commodity Pairs

As a rule, unvalidated residues should not be reported. However, unvalidated residues may be reported on a case-by-case basis. For example, identification and tentative quantitation of a compound not currently included in the analytical screen or preliminary results for special projects. Procedures to be followed in these instances are as follows:

1. Concentrations less than the estimated LOD shall not be reported.

2. A concentration value is not required when a pesticide is detected at or above the determined LOD and below the determined LOQ. The laboratory must code the finding as "Q" [residue at below quantifiable level (BQL)] in the "Annotated Info." section of the LIF and provide the estimated LOD and LOQ concentrations. The concentration will be converted to ½ LOQ in the PDP database for reporting purposes. The laboratory shall enter "HU" (standard not in matrix – unvalidated residue)

or "MU" (standard in matrix - unvalidated residue) in the quantitation field of the LIF.

3. Residue concentrations greater than or equal to the estimated LOQ shall be reported on the LIF. The laboratory shall enter "HU" (standard not in matrix – unvalidated residue) or "MU" (standard in matrix – unvalidated residue) in the quantitation field of the LIF.

4. Results for unvalidated non-detects shall be coded as "U" in the quantitation field of the LIF.

5.2 Reporting Non-violative results

Non-violative results for PDP reporting purposes are residue determinations that do not exceed a stated tolerance. A tolerance is the maximum amount of a pesticide residue that is permitted in or on a food. A detected residue concentration is considered to be non-violative if it is equal to or less than the 40 CFR 180 tolerance for the given commodity. If no commodity tolerance exists then the group tolerance (if available) should be used. If no commodity or group tolerance is established or section 18 reference noted, the tolerance shall be considered zero. All concentrations shall be reported on the LIF.

5.3 Reporting A Presumptive Tolerance Violation (PTV)

Tolerances are established by EPA under the authority of the Federal Food Drug and Cosmetic Act (FFDCA) and are listed in 40 CFR 180. Tolerances are usually established for a specific commodity, however, tolerances may also be established by the commodity groupings established by EPA in 40 CFR 180 or Section 18 tolerances may apply.

a. A residue is considered to exceed the 40 CFR 180 tolerance when the reported value exceeds the tolerance by one number in the second significant figure, or in the case of a single significant figure in the tolerance expression, by one number in that significant figure. For example, if the tolerance is 20 ppm, then a "presumptive violation" would occur at 21 ppm. If the tolerance is 1.0 ppm, then a "presumptive

violation" would occur at 1.1 ppm. If the tolerance is 1 ppm, then a "presumptive violation" would occur at 2 ppm.

b. If the pesticide residue does not have an established tolerance, the laboratory shall report the appropriate code in the annotated information field of the LIF (refer to attachment 01).

c. PTV Notification Policy

All PTVs will be transmitted via RDE. MPO will notify HQ FDA. If States have a cooperative agreement with local FDA, MPO will also send a State-specific report to the laboratories, if requested.

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ELECTRONICALLY REPRODUCED SIGNATURES

Revision 8	May 2003	PDP QA/Technical Meeting

- Updated references
- Updated procedures for reporting validated and unvalidated residues to reflect current database codes and definitions

Pesticide Data Program
Laboratory Information Form Codes

CONFIRMATION CODES	
CODE	CONFIRMATORY METHOD
A	Atomic Emission Detector (AED)
C	Alternate Column
CD	Alternate Column and Alternate Detector
D	Alternate Detector
F	Fluorescence Detector
HR	High Resolution MS
I	Ion Trap Detector (ITD)
IA	Immunoassay
L	Liquid Chromatography w/ Mass Spectrometer (LC/MS)
LT	LC-MS/MS
M	Mass Selective Detector (MSD)
MO	Quantitation & Confirmation by MSD only
P	Alternate Mobile Phase (AMP)
R	Diode Array Detector (DAD)
S	MS as Alternate Detector
T	Tandem Mass Spectrometry (MS/MS)
Z	Other

ANNOTATION CODES	
CODE	ANNOTATED INFORMATION
Q	Residue at Below Quantifiable Level (BQL)
QV	Residue at <BQL> with a Presumptive Violation - No Tolerance
QX	Residue at <BQL> with a Presumptive Violation - Exceeds Tolerance
V	Residue with a Presumptive Violation - No Tolerance
X	Residue with a Presumptive Violation - Exceeds Tolerance

QUANTITATION CODES	
CODE	QUANTITATION
H	Standard Not in Matrix
HU	Standard Not in Matrix - Unvalidated Residue
M	Standard In Matrix
MU	Standard In Matrix - Unvalidated Residue
PH	Standards Prepared Using Analyte Protectants - Not in Matrix
PM	Standards Prepared Using Analyte Protectants - In Matrix
SH	Internal Standards - Not in Matrix
SM	Internal Standards - In Matrix
U	Non-detect - Unvalidated Residue

Pesticide Data Program
Laboratory Information Form Codes

MEAN RESULT CODES	
CODE	MEAN RESULT
A	Detect - Average of Original and Re-extraction Analyses Values
FM	Failed Matrix Spike - Water Only
FP	Failed Process Control - Water Only
M	Not Analyzed
ND	Non-Detect - Original Analysis
O	Detect - Original Extraction Value
R	Detect - Re-extraction Analysis Value
U	Unable to Analyze

DETERMINATIVE CODES	
CODE	DETERMINATIVE METHOD
01	GC/ECD - Electron Capture Detector
02	GC/FPD - Flame Photometric Detector in Phosphorus Mode
03	GC/FPD - Flame Photometric Detector in Sulfur Mode
04	GC/ELCD - Electrolytic Conductivity Detector in Nitrogen Mode
05	GC/ELCD - Electrolytic Conductivity Detector in Halogen Mode
06	GC/FID - Flame Ionization Detector
07	GC/MSD - Mass Selective Detector
08	GC/ITD - Ion Trap Detector
09	TLC - Thin Layer Chromatography
10	HPLC - Liquid Chromatography w/ FL Detector
11	HPLC - Liquid Chromatography w/ UV Detector
12	HPLC Post-Column Derivatization w/FL Detector
14	GC/NPD - Phosphorus Mode
15	GC/NPD - Nitrogen Mode
16	GC/NPD - Nitrogen/Phosphorus Detector
18	GC/FPD - Flame Photometric Detector in Nitrogen Mode
19	HPLC - Pre-Column Derivatization W/FL Detector
27	GC/AED - Atomic Emission Detector
28	AED - Element Selective
30	GC/ELCD - Electrolytic Conductivity Detector in Sulfur Mode
34	GC with Ion Trap MS/MS
58	GC - Gas Chromatography w/ Detector other than Listed
59	HPLC - Liquid Chromatography w/ Detector other than Listed
60	Halogen Specific Detector (XSD)
61	LC/MS - Liquid Chromatography w/ Mass Spectrometer
62	LC/MS/MS - Liquid Chromatography w/ Tandem Mass Spec
63	Second LC/MS
64	Second LC/MS/MS
65	GC/Micro ECD - Micro Electronic Capture Detector
66	GC/PFPD - Pulsed Flame Photometric Detector
67	Third LC/MS/MS
68	Second GC/ECD
98	Immunoassay Screen
99	OTHER

Pesticide Data Program
Laboratory Information Form Codes

EXTRACTION CODES	
CODE	EXTRACTION METHOD
000	No Extraction Necessary
015	Modified Luke Extraction Method without Cleanup for Multi-Residues & Carbamates
550	CDFA Lee et al C-18 Extraction Method
551	CDFA Chlorinated ACN Florisil SPE Extraction Method
552	CDFA Carbamate C-18 Extraction Method
553	CDFA Carbamate SPE Extraction Method
800	FL-Modified CDFA C-18 Extraction Method (P-fraction)
801	FL-Modified CDFA C-18 Extraction Method Aminopropyl SPE Cleanup
802	FL-Modified CDFA C-18 Extraction Method w/ Florisil SPE Cleanup
803	GIPSA Modifed Method for Extraction of Multi-Residues in Grains
804	GIPSA Modified Method for Determination of Triazole Metabolites in Wheat Flour
805	MDA Modified Quecher's Method
806	NYS Modifed SPE Method (F&V)
807	NYS Modified Method for Determination of Triazoles and Metabolites in Peaches
808	WSDA Modified Method for Determination of Triazoles and Metabolites in Apples
809	NSL Butter Extraction Method
810	Montana SPE Triazole Extraction Method for Water
811	Montana SPE Extraction Method for Polar Pesticides (Water)
812	Montana Liquid/Liquid Extraction Method for Non-Polar Pesticides
813	NSL Milk Extraction Method
900	Liquid/Liquid Method
901	NYS Modification of USGS Method 2001/2002 (SPE, GC)
902	NYS Modification of USGS Method 9060 (SPE, LC)
903	NYS Modification of USGS Method for Chloroacetanilide Metabolites (SPE, LC)
998	OTHER Single-Analysis Methods
999	OTHER Multi-Residue Methods